Sept. 30, 1952 F. A. ANGONA 2,612,036

APPARATUS FOR MEASURING INTERSTITIAL WATER CONTENT OF WELL CORES

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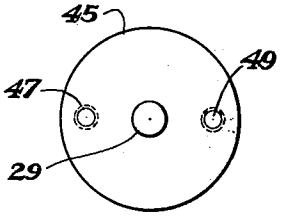


Fig. 3

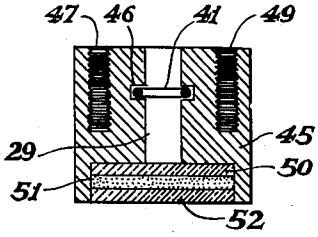


Fig. 2

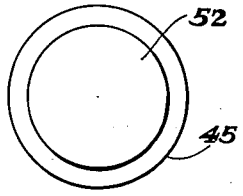


Fig. 4

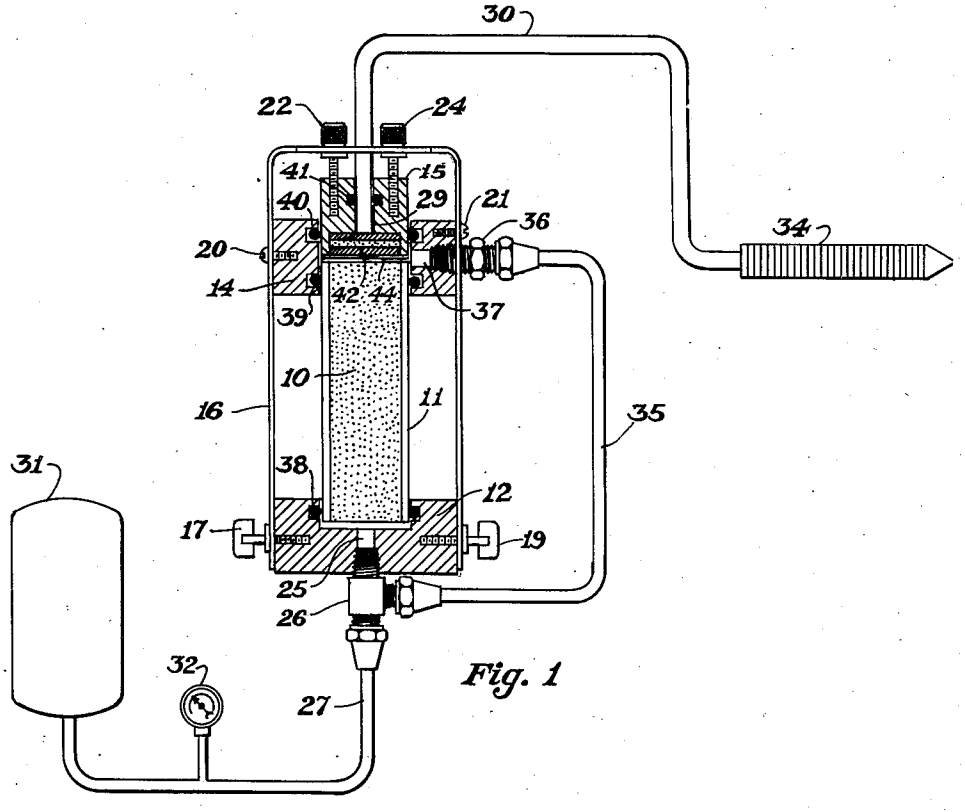


Fig. 1

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APPARATUS FOR MEASURING INTERSTITIAL WATER CONTENT OF WELL CORES

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4 Claims. (Cl. 73—38)

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This invention relates to semi-permeable membranes and relates more particularly to semi-permeable membranes for use in measuring the interstitial water content of porous materials of terrestrial origin, particularly samples of cores taken from underground formations encountered in the drilling of wells for petroleum.

It is customary in the drilling of wells for petroleum to remove samples or cores from the various formations encountered and to test the cores for various physical characteristics and properties. One of the characteristics of the cores usually determined is the interstitial, or as it is sometimes termed, the connate, water content of the cores. In order to minimize errors which may be encountered as a result of evaporation of water from the cores or as a result of water entering the core from the drilling fluid or mud employed during the drilling of the well, it has been proposed to determine the interstitial water content of the cores by a method known as the "restored state method." In accordance with this method, the cores are subjected to conditions simulating those under which they existed in their original terrestrial positions and their water contents measured when at equilibrium under these conditions. In practicing this method, the core is maintained in contact with a body of free water and a pressure of a water immiscible gas or liquid imposed upon the core. The value of this pressure is such that the difference in pressure between the core and the body of free water approximates the pressure differential between the interstitial water of the core when in its original position in the earth and the water table underlying the core, and this pressure differential is maintained until the water content of the core reaches equilibrium. This method is also employed for determining the "minimum interstitial water content" of the core, i. e., the water content which remains substantially constant with increase in the pressure differential.

In order to prevent flow of the pressure inducing medium into the body of free water and thereby to maintain the pressure differential between the core and the body of free water, a semi-permeable membrane saturated with water is interposed between the core and the body of free water. This semi-permeable membrane may be defined as a membrane which, when saturated with one fluid, has a minimum displacement pressure to another fluid immiscible with the first. Stated otherwise, the membrane, when saturated with a fluid such as, for example, water, will

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be permeable to the flow of water but will be impermeable to the flow of another fluid, such as a gas or liquid, immiscible with the water, until a certain minimum pressure of this other fluid has been exceeded. Thus, by interposing the semi-permeable membrane between the core and the body of free water, the fluid imposing a pressure on the core will be unable to permeate through the membrane into the body of free water and thereby the pressure differential can be maintained. Further, water will be able to flow to or from the core and the body of free water through the membrane until the water content of the core reaches equilibrium under the pressure differential imposed.

Various types of materials have been employed for the semi-permeable membrane. For example, consolidated materials such as unglazed porcelain or fire clay or unconsolidated materials such as barium sulfate have been employed. However, the use of these materials have been attended with certain difficulties. Consolidated materials have low minimum displacement pressures. Unglazed porcelain for example, when saturated to water, will be permeable to air or nitrogen at pressures of about 15 pounds per square inch gage. Membranes made from unconsolidated materials are not easily handled, and are readily disturbed or broken by repeated removal and contacting of the core where the attainment of equilibrium water content is determined by weighing the core. Further, where liquids are employed for imposing a pressure on the core, membranes made from unconsolidated materials tend to be disturbed or broken by the buoyancy effect of the liquid.

It is an object of this invention to provide a semi-permeable membrane. It is another object of this invention to provide a semi-permeable membrane having a high displacement pressure. It is another object of this invention to make possible the measurement of interstitial water at high pressure differentials. These and other objects of the invention will become apparent from the following description thereof.

The semi-permeable membrane of the invention consists essentially of a layer of unconsolidated material positioned between two layers of consolidated material. In the preferred form of the semi-permeable membrane, the two layers of consolidated material and the layer of unconsolidated material are maintained in contact by supporting means in order that the mem-

brane be readily handled without the layers coming apart.

As consolidated material, any type of porous material heretofore employed for semi-permeable membranes may be employed. The essential characteristics of such materials are that they be porous and, when saturated with one fluid, have a minimum displacement pressure to another fluid immiscible with the first. Suitable types of materials are unglazed porcelain, fire clay, ultra-fine fritted glass, pumice stone, etc.

As unconsolidated material, any type of material of small particle size may be employed provided such material is not water soluble. It is preferable also that the material be substantially non-swelling in the presence of water. Suitable types of material are barium sulfate, powdered silica, and powdered pumice. Preferably, however, talc is employed.

The semi-permeable membrane of the invention has a high displacement pressure. The displacement pressures vary with the type of liquid employed for saturating the membrane, whether plain water or saline solution, and with the fluid employed for imposing the pressure differential. The displacement pressures also vary with the type and particle size of the unconsolidated material and with the type of consolidated material. In any case, however, the displacement pressure will be considerably higher than the displacement pressure of the unconsolidated material or the consolidated material alone. For example, where unglazed porcelain and talc, the talc having a particle size between 0.18 and 4.5 microns, are employed and the membrane is saturated with water, the displacement pressure to air or nitrogen will be about 40 pounds per square inch gage.

In the copending application of Paul P. Reichertz, Serial No. 766,566, filed August 6, 1947, now Patent No. 2,539,355, issued January 23, 1951, there is disclosed and claimed a method and apparatus for measuring, among other characteristics, the interstitial water content of cores. The semi-permeable membrane of the present invention is highly useful when employed in the apparatus of the copending application. The following more detailed description of the semi-permeable membrane of the present invention will be given in connection with its use in the apparatus of the copending application.

Fig. 1 is a sectional view of apparatus containing the semi-permeable membrane for measurement of interstitial water of well cores.

Fig. 2 is a sectional view of a plug member containing the semi-permeable membrane.

Fig. 3 is a plan view of the top of the plug member of Fig. 2.

Fig. 4 is a plan view of the bottom of the plug member of Fig. 2.

Referring now to Fig. 1, a cylindrical well core 10, covered along its outer edges with a coating 11 of an impervious material, such as formaldehyde phenol resin ("Bakelite"), methyl methacrylate resin ("Lucite"), or polystyrene, is positioned between a lower annular base member 12 and an upper annular base member 14. Positioned in the upper base member 14 and adjacent to the upper surface of the core 10 is a plug member 15. Maintaining the base members 12 and 14 and the plug member 15 in spaced relationship is a rigid U-shaped strap 16. Base member 12 is held on the strap by means of wing bolts 17 and 19 and base member 14 is held on the strap by means of screws 20 and 21. Tightening screws 22 and 24 hold plug member 15 on the strap and also serve the function of tightening the plug member into close contact with the upper surface of the core 10.

Leading through the base member 12 is channel 25 connecting through fitting 26 to pipe 27. Leading through plug member 15 is channel 29 through which pipe 30 is fitted. Pressure chamber 31 and pressure indicator 32 are connected to pipe 27 and measuring burette 34, preferably positioned level with the top of core 10, is connected to pipe 30. To provide the same pressure at both the bottom and top of core 10, pipe 35 leads from fitting 26 to fitting 36 connecting with channel 37 in upper base member 14. To prevent leakage, gasket 38 is provided in base member 12, gaskets 39 and 40 are provided in base member 14, and gasket 41 is provided in plug member 15.

Plug member 15 contains semi-permeable membrane 42. The membrane is open at its upper surface to channel 29 and pipe 30, and is adjacent at its lower surface to the exposed top of core 10. To provide a close capillary contact of the lower surface of the semi-permeable membrane with the exposed surface of the core 10, a thin sheet of porous material 44, which may be closely woven cloth or tissue paper, is placed between the core and membrane. In Fig. 1, the core, the sheet of porous material 44, and the plug member 15 are shown as being slightly separated. However, this separation is only for purposes of clarity in illustration. In operation, as mentioned hereinafter, the core 10, the sheet of porous material, and the plug member 15 are maintained in close contact by means of tightening screws 22 and 24.

To determine interstitial water content of a well core, the core is first leached with acetone, benzene, toluene or other suitable solvent to remove oil or other liquid material naturally contained therein and then dried of solvent. The core is next covered along its outer edges with an impervious coating of methyl methacrylate resin ("Lucite"), formaldehyde phenol resin ("Bakelite"), or polystyrene and weighed. Following this, the core is completely saturated with water or with an aqueous solution having the same physical or chemical properties of the liquid contained in the core in its original terrestrial position. The core is again weighed. From the difference in weights, the total pore volume of the core may be calculated knowing the density of the water or solution. The semi-permeable membrane is also saturated with the same liquid employed for saturating the core.

The core is positioned as shown in Fig. 1 and the pipe 30 is filled from the top of the membrane to at least the zero mark on the measuring burette with the same liquid employed for saturating the core. The desired pressure, which may be that of a gas such as air or nitrogen or a liquid immiscible with the liquid employed for saturating the core, is then impressed on the core from pressure chamber 31 as measured by pressure indicator 32. Immediately after imposition of the pressure, the plug member 15 is tightened downwardly by means of tightening screws 22 and 24 to effect a close capillary contact between the membrane 42, the porous sheet 44, and the exposed surface of the core 10. Upon imposing the pressure and tightening the plug member, there is an initial rapid flow of excess liquid to the measuring burette 34 and, when this initial rapid flow of excess liquid stops, a reading on the burette 34 is made.

As a result of capillary forces induced by the

pressure, liquid is removed from the core and passes through the membrane 42 to the pipe 30 and the burette 34. This pressure is maintained until the liquid content of the core under the pressure imposed comes to equilibrium. Attainment of equilibrium is a slow process and may require a period of time as long as thirty days. When equilibrium is attained, a second reading is made on the burette and the difference between the two readings will be the volume of liquid removed from the core. The interstitial water content of the core at the pressure employed will be the difference in the volume of liquid in the core at saturation and the volume of liquid removed and may be expressed as the percentage of total pore volume occupied by the liquid.

Figs. 2, 3, and 4 illustrate the plug member 15 and the semi-permeable membrane 42 in greater detail. The plug member 15 consists of a body portion 45 containing channel 29 provided with recess 46 for gasket 41. Fitted into the body portion are threaded bushings 47 and 49 adapted to receive tightening screws 22 and 24. Preferably, the body portion is a plastic material such as methyl methacrylate resin ("Lucite"), formaldehyde phenal resin ("Bakelite"), or polystyrene. Fitted into the body portion is the semi-permeable membrane comprising a layer of consolidated material 50, a layer of unconsolidated material 51, and another layer of consolidated material 52.

To fabricate the semi-permeable membrane, a suitably sized cylinder of plastic material, such as methyl methacrylate resin, is made as, for example, in a mold press. A hole having the same diameter as the membrane to be fitted is then drilled into the cylinder to form an annulus. The annulus is returned to the mold press, one end of the annulus being positioned flush with a smooth plane surface of the press. The disc of consolidated material 52 is fitted into the annulus and the layer of unconsolidated material 51 placed on top of the disc of consolidated material. The layer of unconsolidated material is packed tightly in position and the disc of consolidated material 50 placed over the layer of unconsolidated material. The remaining portion of the annulus is filled with methyl methacrylate powder and the mold press closed. The pressure in the mold is brought to 200 pounds per square inch and the temperature is increased gradually to 275° F. When this temperature is attained, the pressure is increased to 5000 pounds per square inch, following which the mold is permitted to cool to room temperature. The cooled cylinder is removed from the mold and the channel 29 is drilled therein. The recess 46 is then machined in the channel and the gasket 41 fitted into position. Finally, holes are drilled to accommodate the threaded bushings 47 and 49 and the bushings fitted therein.

While I have thus described my invention, it is to be understood that such description has been given by way of illustration and example only, and not by way of limitation, reference for the latter purpose being had to the appended claims.

I claim:

1. In an apparatus for determining interstitial water content of a well core sample by the restored state method, said apparatus having a chamber for holding said well core sample, means for supporting a semi-permeable membrane in contact with said well core sample, means for imposing upon said well core sample a pressure of a non-aqueous fluid, and means providing a conduit leading to said semi-permeable membrane for passage of fluid, a semi-permeable membrane comprising two unitary disks of unglazed porcelain and a layer of powdered talc positioned between and in contact with said disks of unglazed porcelain.

2. In an apparatus for determining interstitial water content of a well core sample by the restored state method, said apparatus having a chamber for holding said well core sample, means for supporting a semi-permeable membrane in contact with said well core sample, means for imposing upon said well core sample a pressure of a non-aqueous fluid, and means providing a conduit leading to said semi-permeable membrane for passage of fluid, a semi-permeable membrane comprising two unitary disks of unglazed porcelain and a layer of powdered talc of particle size between 0.18 and 4.5 microns positioned between and in contact with said disks of unglazed porcelain.

3. In an apparatus for determining interstitial water content of a well core sample by the restored state method, said apparatus having a chamber for holding said well core sample, means for supporting a semi-permeable membrane in contact with said well core sample, means for imposing upon said well core sample a pressure of a non-aqueous fluid, and means providing a conduit leading to said semi-permeable membrane for passage of fluid, a semi-permeable membrane comprising two unitary disks of fire clay and a layer of powdered talc positioned between and in contact with said disks of fire clay.

4. In an apparatus for determining interstitial water content of a well core sample by the restored state method, said apparatus having a chamber for holding said well core sample, means for supporting a semi-permeable membrane in contact with said well core sample, means for imposing upon said well core sample a pressure of a non-aqueous fluid, and means providing a conduit leading to said semi-permeable membrane for passage of fluid, a semi-permeable membrane comprising two unitary disks of fire clay and a layer of powdered talc of particle size between 0.18 and 4.5 microns positioned between and in contact with said disks of fire clay.

FRANK A. ANGONA.

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